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# Lubrication

## A Technical Publication Devoted to the Selection and Use of Lubricants

*This Issue*

**MICROCHEMISTRY IN LUBRICATING OIL ANALYSIS**

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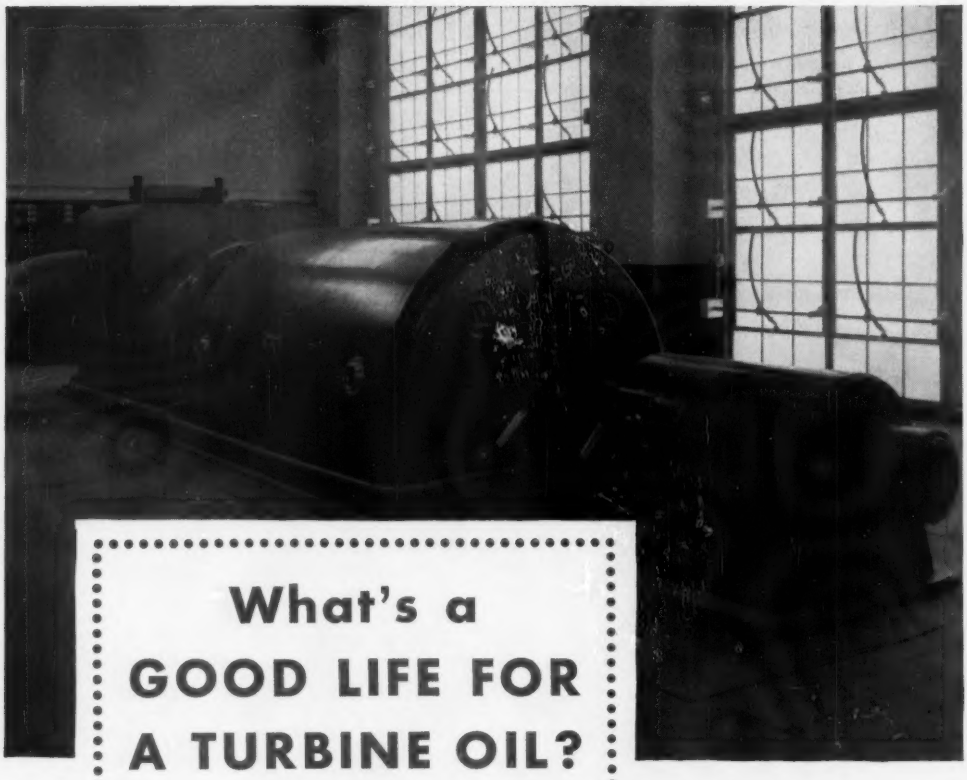
TEXACO PETROLEUM PRODUCTS



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# LUBRICATION

A TECHNICAL PUBLICATION DEVOTED TO THE SELECTION AND USE OF LUBRICANTS

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# Microchemistry In Lubricating Oil Analysis

MICROANALYSIS is a relatively new branch of science and, like many other developments, was born out of necessity. At the University of Graz, Austria-Hungary, the biochemist Pregl labored for years on gallstone research. Finally in 1910 he reached a crucial stage in his experiments. Before him were a few small crystals, whose composition it was necessary to know before he could continue his research. He could spend several years repeating his experiments on a larger scale or he could devise some new techniques to make suffice the small sample available for analysis. He chose the latter course and thus became one of the pioneers in the field of microanalysis. Pregl became so fascinated by his new field of endeavor that he never returned to his original research but embarked on a new career that won him the Nobel Prize in 1923.

Pregl worked in organic chemistry. Simultaneously Emich, another Austrian, applied microanalysis to inorganic chemistry. Although several workers had practiced microanalysis previously, especially using the microscope, Pregl and Emich are generally credited with being the founders of microchemistry.

In the United States, between 1920 and 1930, a few colleges and universities added microanalytical laboratories to their chemistry departments. In many cases these laboratories were headed by former students of Emich or Pregl. The pharmaceutical companies were the first industrial concerns to embrace microanalysis since small samples are common in biological work. Much of the work done on vitamins and similar materials isolated from plant and animal products would have been impossible without microchemical techniques.

That the petroleum industry which measures its yield of product in terms of millions of barrels should have need of microchemical methods of analysis would seem strange indeed. However, it happened in ever increasing numbers of cases that the petroleum analyst was forced to report "sample too small for analysis" on the test report instead of the desired test results. In a great number of these instances the information which could not be obtained was considered valuable. The loss of this sometimes valuable information prodded the petroleum industry into an awareness of the worth of microanalysis. Today no major petroleum laboratory can be considered complete without a well-equipped microchemical laboratory.

## MICROCHEMICAL TECHNIQUES

The term microanalysis is normally applied to techniques handling sample quantities of 10 milligrams or less. The petroleum industry, using a much broader interpretation, defines microanalysis as applying to the analysis of samples that are of too limited a quantity to be analyzed by standard procedures. This does not mean that microanalysis, in the more limited sense, is not used as such by the

petroleum industry but rather that as a matter of convenience one term is used to replace many. Thus, miniaturized semi micro, micro and ultra micro procedures are all grouped together as micro procedures.

Microanalytical methods use the metric system for all measurement. The following illustrates the relative quantities involved:

| | | |
|---|---|---|
| milligram | $= \frac{\text{gram}}{1{,}000}$ | A five cent piece weighs about 5 grams |
| microgram | $= \frac{\text{gram}}{1{,}000{,}000}$ | Also called a gamma, $\gamma$ |
| milliliter | $= \frac{\text{liter}}{1{,}000}$ | About 20 drops or 1 gram of water |
| microliter | $= \frac{\text{liter}}{1{,}000{,}000}$ | A microliter of water weighs about 1 milligram |

## General Concepts

The basic idea of microanalysis is the reduction in size of analytical apparatus to suit small samples. In some cases, reduction in size alone has sufficed; more often, retention of the original form of the apparatus is unsatisfactory and new principles must be employed. As in regular scale analysis, microanalysis is dependent on transforming the unknown materials into one or more known materials. Thus it is necessary to separate, purify, and measure the quantities of materials obtained from the unknown. Among the operations that are performed in microanalysis are weighing, measurement of volume, solution, filtration, washing, evaporation, drying, ignition, distillation and other operations familiar in regular scale analysis.

# CHEMICAL TESTS

## Ash Content

There are primarily two ways in which microchemistry is useful in determining ash content. One instance is when only a small amount of sample is available for ash determination. Micro-size crucibles are used to hold the sample and a micro balance is used to make the weighings. Special micro muffle furnaces have been constructed to provide even heat distribution. Their small size greatly reduces construction costs and permits a more generous use of quartz and platinum linings. There are other cases in which a large amount of sample is available, but the ash formed may represent only a trace of the original sample. In these instances, macro equipment is used and macro quantities of sample are processed, but the ash residue must be weighed on a micro balance to provide the necessary accuracy.

## Neutralization and Saponification Number

The micro modifications for neutralization and saponification number are essentially scaled down versions of the macro procedure. In order to obtain an accurate neutralization or saponification number on a micro sample of oil, it is necessary to be able to measure very small volumes of reagents very accurately. The use of an ultra-burette that can measure as little as one-thousandth of a milliliter with certainty is essential.

## Elemental Organic Analysis

*Carbon and Hydrogen*

Since most organic compounds contain carbon and hydrogen their determination is the most frequently performed elemental organic analysis. The method usually employed is to burn the sample in oxygen which converts the carbon to carbon dioxide and the hydrogen to water. These two products of combustion are collected by absorbents and weighed. In micro procedures using ten milligrams or less, the combustion proceeds quite rapidly. For this reason such micro methods are preferable to macro methods even when large amounts of sample are available.

*Oxygen*

A basic concept of microanalysis often implied but seldom expressed is that the apparatus design should incorporate as many of the necessary micro manipulations and chemical steps as possible, i.e., (built-in intelligence) freeing the chemists from as many of the detailed steps as is practical. The microchemical determination of oxygen in organic compounds using the method first developed by Schütze and modified by Untezaucher is a good example of this principle. In essence, the sample is heated in an atmosphere of nitrogen. The oxygen of the sample forms carbon monoxide which is oxidized to carbon dioxide which is absorbed and weighed. The actual apparatus is complex since it is designed to accomplish all the intermediate reactions necessary to produce the carbon dioxide in a pure form for weighing. Figure 3 is a diagram of one form of the apparatus.

*Nitrogen*

In the Dumas Method for the micro determination of nitrogen the sample is burned at red heat with copper oxide to yield nitrogen and oxides of nitrogen. The latter are reduced to nitrogen by a section of finely divided copper. An atmosphere of pure carbon dioxide is used to sweep the nitrogen from the combustion tube and into aqueous potassium hydroxide solution where the $CO_2$ is absorbed. The volume of nitrogen is read at atmospheric pressure and its weight is calculated from the volume, pressure, and temperature. This method is a good example where a micro method is preferable regardless of the amount of sample available for analysis.

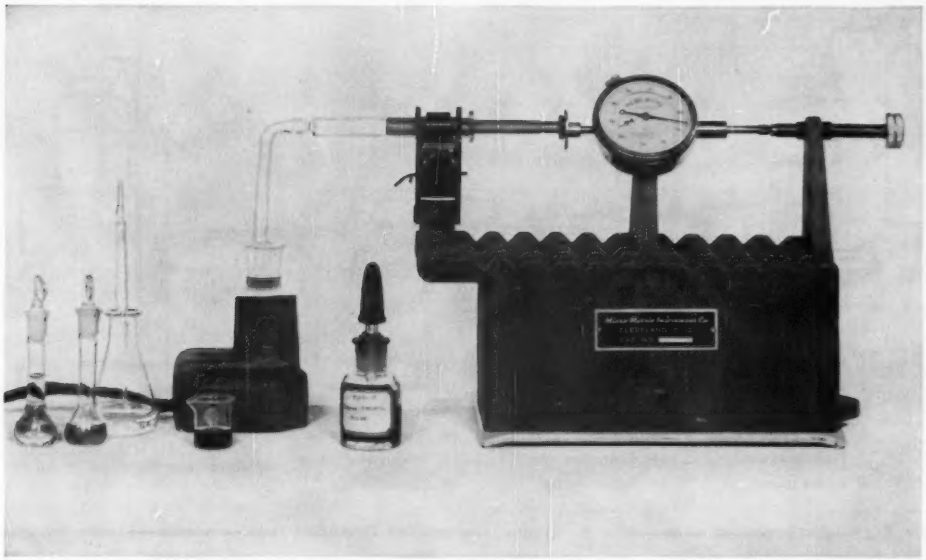


Figure 1 — Titration on a micro scale using a micro magnetic stirrer and a micro burette capable of delivering as little as 1/1000 milliliter of reagent.

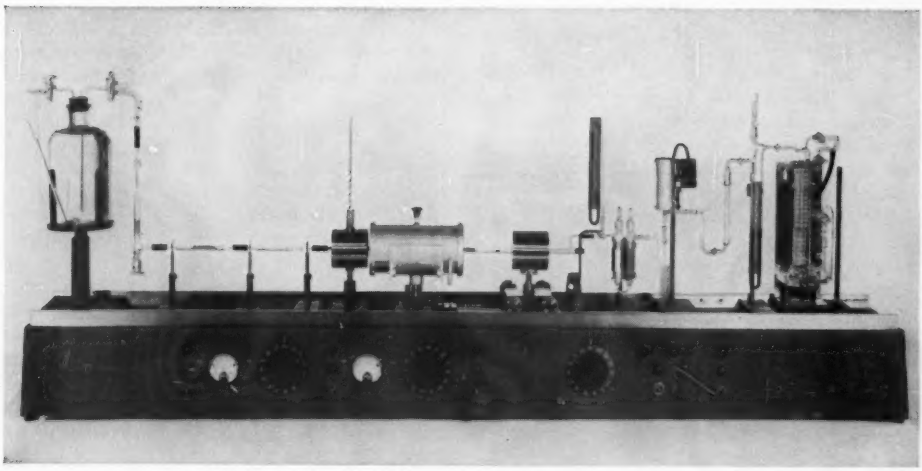
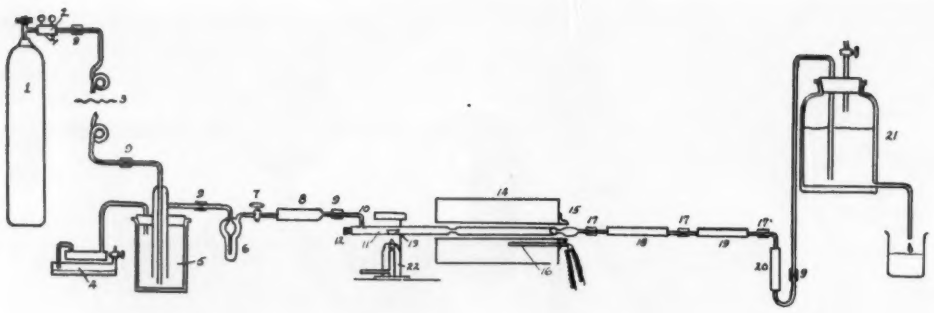


Figure 2 — Combustion train for micro carbon hydrogen determination.



1. Nitrogen cylinder
2. Reduction valve
3. Glass spiral
4. Excess pressure vessel
5. Pressure regulator
6. Bubble counter
7. Stopcock
8. Gas purifying tube
9. Rubber connection
10. Side arm
11. Combustion tube
12. Rubber stopper
13. Boat
14. Electric furnace
15. Tube support
16. Thermocouple tube
17. Impregnated tubing
18. Oxidation tube
19. Micro absorption tube
20. Drying tube
21. Mariotte flask
22. Burner with chimney and roof

Figure 3 — Schematic drawing of apparatus for oxygen determination illustrating complexity of some micro equipment.

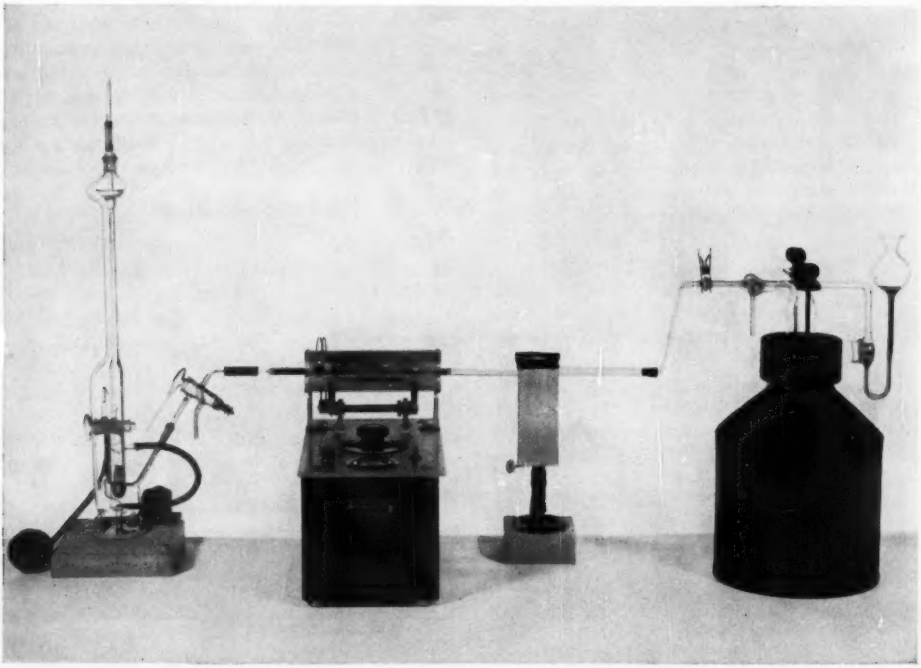


Figure 4 — Micro Dumas apparatus for nitrogen determination.

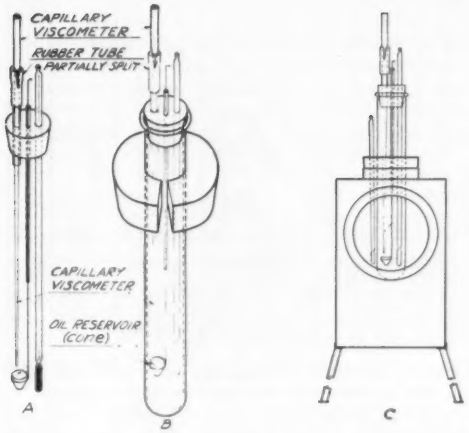



Figure 5 — Micro apparatus for determining the viscosity of a liquid by measuring the influx time of the liquid in a narrow capillary tube.

*Sulfur, Halogens and Phosphorus*

Sulfur, halogens and phosphorus in organic compounds are determined by heating the sample with fuming nitric acid in sealed glass tubes in a Carius oven. When this method of destroying the organic material is used on a micro sample, the danger due to pressure built up in the Carius tubes is kept to a minimum. In micro procedures for sulfur, halogens and phosphorus, it is necessary to collect, filter and weigh micro quantities of precipitates. It is obvious that the usual folded paper cone cannot be satisfactorily reduced in size to permit the removal and recovery of a small precipitate from a small volume of liquid. Micro-filtration may be accomplished in a number of ways, but the most convenient is to draw the liquid into a capillary pipette provided with a retaining wall which holds a very small pellet of the filtering medium. In this way a small volume of liquid can be filtered, and the precipitate washed, dried and weighed.

## Molecular Weight

The microdetermination of the molecular weight of a substance may be accomplished by several different methods, most of which are small-scale applications of the various macromethods found throughout the literature, such as elevation of boiling point, depression of the freezing point or melting point, and vaporimetric methods. A method that is singularly adapted to micro molecular weight determination is the diffusion procedure of Barger. If two solutions having a common solvent are placed in an evacuated system and held at a constant temperature, the solvent will distill from the solution of greater vapor pressure to the one of the lesser, until equilibrium is reached. Then the volumes of the two solutions will be constant and equimolar. If one solution contains a known substance and the other solution contains an unknown, the molecular weight of the latter can be calculated from the data obtained in such an experiment.

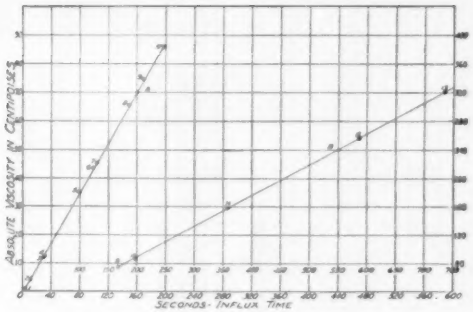



Figure 6 — The relationship between the influx time and the absolute viscosity.

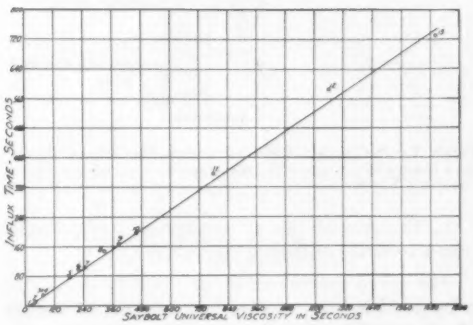



Figure 7 — The relationship between the influx time and the Saybolt Universal viscosity.

# PHYSICAL TESTS

## Specific Gravity

The specific gravity of a lubricating oil may indicate, when other properties such as viscosity are known, the type of base oil from which the lubricating oil was made. The specific gravity or density can be determined on less than 0.1 milliliter of oil by using a micro pycnometer made by sealing off a 0.1 milliliter pyrex pipette having 0.01 milliliter subdivisions. If the volume of oil is measured at the desired temperature and its weight determined by a semi-micro balance, it is possible to determine its density to $\pm 0.003$ grams per milliliter.

## Viscosity

Viscosity is the most important single property of a lubricating oil. Many different methods for microviscosity have been devised, but they can be divided into the following categories based on the principles used in the microviscosimeter.

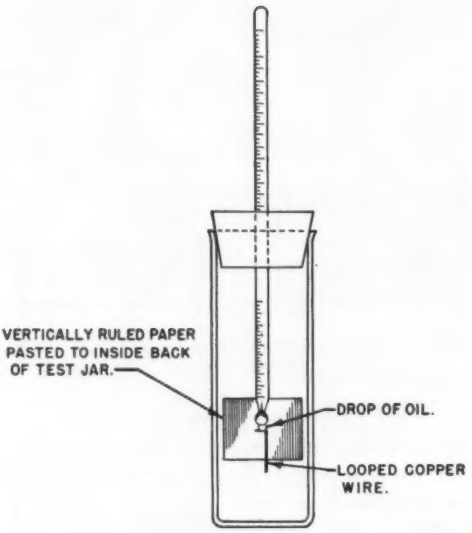



Figure 8 — Micro pour point apparatus. The looped copper wire is used to indicate when the drop of oil has become rigid.

1. The rate of rise of the meniscus of a liquid into a vertical capillary. (Figure 5).

The micro determination of viscosity using this principle has been applied to a fraction of a drop of oil. A capillary tube is lowered into the oil sample held in a metal cone reservoir and at the same instant a stop-watch is started. The time required for the oil to rise in the capillary to an arbitrarily placed line is measured. By reference to a curve based on data obtained with samples of known viscosity, the kinematic viscosity in centistokes can be obtained. Since the surface tension of the oil is a factor in this determination, the unknown should be compared to a curve for a similar type of oil.

2. The efflux time of a constant volume of liquid. This type of microviscosimeter is similar to the Saybolt type viscosimeter.

3. The rate of fall of a steel ball in a liquid. The microviscosimeters of this type are miniaturized versions of macro falling sphere viscosimeters. If one measures the rate of fall of a sphere in a liquid, the absolute viscosity of the liquid can be calculated from Stokes' Law.

4. The rate of fall of the meniscus of a liquid previously drawn up into a vertical capillary. This method is similar in principle to that of kinematic viscosity equipment used in macro scale analysis.

5. The rate of fall of a short segment of liquid in a vertical capillary.

Most of this type of microviscosimeters have an arrangement to vary the angle of inclination of the capillary tube to give a suitable efflux time with oils of varying viscosity.

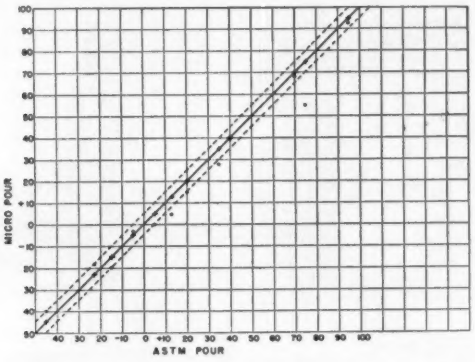



Figure 9 — Correlation between the micro pour and ASTM pour is shown to be very good for most oils tested.

6. Ball and Cup Viscosimeter

The force required to separate a metal ball and cup coated with an oil is a measure of the absolute viscosity of that oil.

## Pour Point

By a simple modification of the well known practice of determining solidification or melting point of wax on a thermometer bulb, it is possible to obtain on a drop of oil pour test results that agree very well with those obtained by the conventional ASTM method using some 30 to 40 grams of sample. To make a determination, the bulb of a thermometer is dipped into a sample of oil. A copper wire with a loop on one end is applied to the drop of oil on the thermometer bulb by its loop. The wire adheres to the drop of oil by surface tension and its stem serves to magnify the movement of the oil drop during pour test observations. The assembly is cooled in the customary ASTM pour test manner until no further movement is observed in the drop of oil when the assembly is removed from the cold bath and held in a horizontal position for 5 seconds. The temperature at which no movement is noticeable is 5°F. below the pour point, as in the ASTM method.

## Color

In the micro ASTM Union color determination of petroleum oils, the glass jar used to hold the sample is similar to that used for the full scale test. However, it has a raised bottom which raises the level of a two ml. oil sample to that level which would be occupied by 30 milliliters of sample if the full scale apparatus were used. The jar is painted black from the top of the raised bottom downward, and from one-eighth inch above the raised bottom upward. This gives a one-eighth inch ribbon-like view across the middle of the viewing window.

### Flash Point

The flash point of an oil can be obtained on less than one milliliter of sample instead of the 70 milliliters required in the Cleveland open cup. The sample is heated in a flash chamber drilled into an aluminum block. The vapors are ignited by a spark directed against the side of the chamber.

### Crankcase Dilution and Water-by-Distillation

Miniaturized versions of the regular size ASTM apparatus for determination of crankcase dilution and water-by-distillation have been built so that these tests can be made on a fraction of the sample size required in the ASTM methods.

## INSTRUMENTAL TECHNIQUES

Many instrumental methods normally use sample quantities of the same order of magnitude as that of micro analytical procedures. While important use of these procedures is made for the analysis of micro samples, micro chemistry cannot lay claim to the methods as such. A short summary of some of the more important instrumental procedures is of value to indicate their place in the micro analytical scheme.

### Microscope

Chemical microscopy is, of course, one of the techniques employed by the professional microchemist. Figure 12 shows him at a specially constructed desk equipped with conventional and binocular microscopes, chemical reagents and auxiliary apparatus. The operation being performed is the electrodeposition of a fraction of a milligram of copper on a tiny electrode fused in a microscope slide. A large portion of the microchemist's work can be performed in this space. The microscopic inspection of minute samples provides the chemist with a great deal of preliminary information which will save him complex and time-consuming examinations.

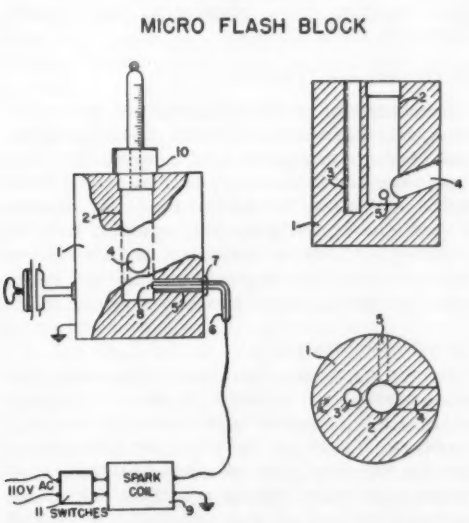



Figure 10 — Micro apparatus for determining flash point of an oil.

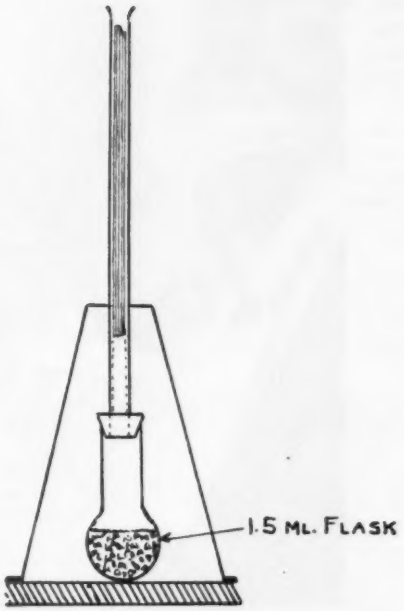



Figure 11 — Apparatus for the detection of anti-freeze in a small sample of used crankcase oil.

Another advantage to be gained by chemical microscopy is that of performing tests in situ. In practice, it is sometimes difficult to isolate physically the particles of material to be studied. For example, it may be desired to learn something of the nature of a tiny inclusion embedded in a metal or a thin film of corrosion product present on its surface. Here again, the microscope is of great service, both in guiding the physical manipulations necessary to restrict the action of the reagents to a localized area and in observing the actual identification reaction (usually chosen to yield an intensely colored product or bubbles of gas).

The micro-manipulator, an instrument designed originally by biologists to perform intracellular oper-

Figure 12 — Micro chemist using a binocular microscope to follow the electro deposition of a fraction of a milligram of copper.

ations, has recently been employed in microchemical work with very satisfactory results. The instrument furnishes the means of regulating with great precision the movement of needles, capillary pipettes, electrodes, electrically-heated platinum wires, etc., under relatively high magnifications. It offers great promise where particles of exceptionally small dimensions are to be studied.

## Emission Spectroscopy

Spot tests that can be run on a fraction of a milligram of sample are available for most of the metals. However, while some of these color reactions are specific for one metal, most of the reagents react with a class of metals rather than one specific metal. Emission spectroscopy can provide both qualitative and quantitative analyses when milligram quantities of metal are present in the sample.

## Infrared Absorption

Infrared absorption is invaluable in the identification of organic compounds, since the infrared absorption pattern of each compound is a unique record in the same manner as the fingerprint of each human is unique. Only a few milligrams of sample are necessary to obtain an infrared absorption. Before this technique was available, identification of an organic compound was a time-consuming operation requiring the preparation of one or more derivatives of the unknown.

## Electrographic Transfer

Sometimes the sample to be analyzed is not only small, but it can not be isolated conveniently mechanically, for example, a faint corrosion film on a metal plate or a contaminating occlusion in a metal surface which may not be destroyed. The analysis of such problems employs electrographic transfer, a technique whereby a trace image of the surface is obtained on special reagent papers which are subsequently subjected to spot analysis for identification.

## Other Instruments

In addition to the instrumental equipment discussed above, the modern petroleum laboratory makes extensive use of other elaborate analytical systems developed by the physicists and physical chemists. Among these are ultraviolet absorption, polarography, mass spectrometry, X-ray diffraction, electron diffraction and electron microscope. Each of these methods is capable of yielding useful analytical information on as little as a milligram of sample.

## Chromatography

The micro chemist has found chromatographic separations to be among the most powerful tools at his disposal. Many separations, all but impossible by other techniques, can be made conveniently using one of the three basic chromatographic methods.

*A. Adsorption Chromatography* uses the molecular adsorption of the active portions of the sample molecules upon a surface active adsorbent such as silica gel, a clay or charcoal. The binding forces are predominently physical with only a small degree of chemical binding. Substances that can be more strongly adsorbed will displace those that are weakly adsorbed. In this way, a gradual stratification of the sample is obtained with the more strongly adsorbed molecules concentrated on the entering portion of the adsorbent. The less strongly adsorbed molecules are displaced and travel further down the column. The adsorbed substances can be removed from the adsorbent by eluting with a solvent which is more strongly adsorbed, displacing the desired component.

This is a valuable tool for separating complex mixtures into individual components that can be identified and measured quantitatively. Additives in oils, oxidation products from used oils, and contaminants can be separated from base oils. The base oils themselves can be separated into paraffinic and aromatic portions.

*B. Partition Chromatography* is based upon the partition of a solute between two solvents, one solvent being fixed by adsorption on a solid phase. This chromatographic process includes both the popular paper chromatographic and gas chromatographic methods. Columnar chromatography using water adsorbed on solid silica gel or a hydrocarbon adsorbed on cellulose is also of great value for micro analysis.

The separations made with this technique are comparable in type with those of the adsorption method. The paper chromatographic method has found great use in the detection, identification and measurement of extremely small and complex samples. It is capable of easily resolving many mixtures that are all but impossible to separate by other means. Paper chromatography is also of great aid in identifying compounds by comparing the relative movement with that of a known sample.

*C. Ion Exchange* is a chromatographic method using chemical bonding forces instead of physical absorption or relative solubility for separating the sample into its various components. The ion exchange resins can be thought of as either acids or bases, depending upon the resin type. The acid or basic radical is held in a sponge-like lattice in which the ions are free to react but are not free to move. In the practical ion exchange resins, the hydrogen ions or hydroxyl ions are less strongly bound by the resin than most other ionic species. This, then, allows a metal or base to be preferentially fixed on the resin displacing less strongly bound ions which are then released to the mobile phase. Most anions and cations have different reactivities; trivalent ions are normally more strongly bound than either divalent or monovalent ions.

Ion exchange is used almost exclusively for inorganic analysis. Metals can be separated from each other and from interfering substances. This enables more accurate determination of the desired elements to be made.

## Radioactivity

Although it has not been used extensively to date, radioactivity holds great promise as a powerful tool in microanalysis. The isotopic dilution technique

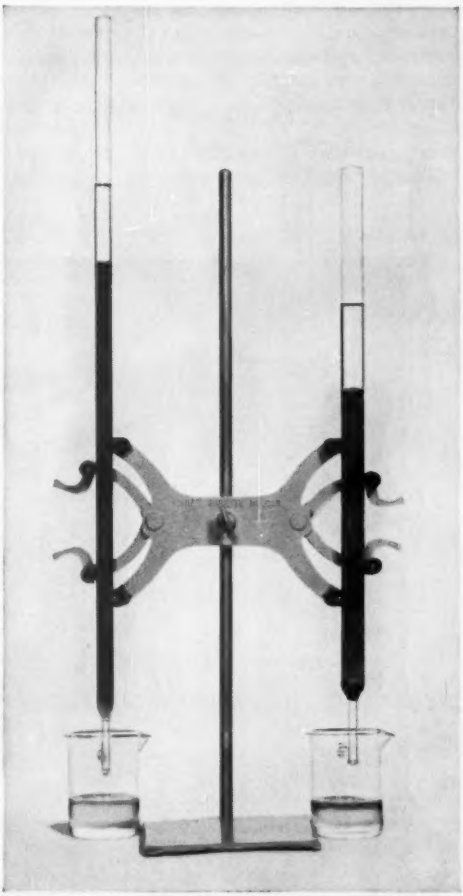


**Figure 13 — Chromatographic columns packed with silica gel used to separate additives from a lubricating oil by preferential adsorption of the additive.**

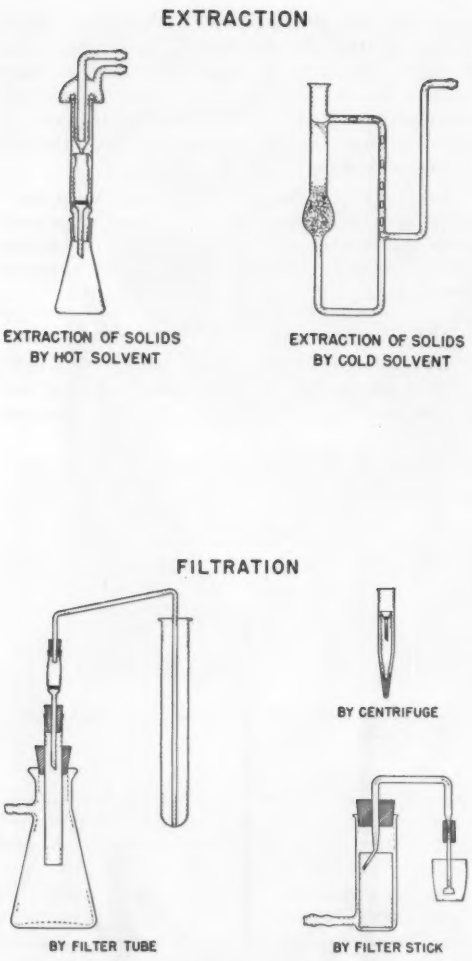



Figure 14 — Equipment used for various separations on a micro scale.

has been used for the determination of gaseous elements such as nitrogen. Instead of attempting to measure a volume of nitrogen as in the Dumas nitrogen determination, the nitrogen obtained from the sample is diluted with a volume of isotopic nitrogen. The ratio of ordinary to isotopic nitrogen can be determined with great precision, and therefore the amount of nitrogen due to the analysis of the sample determined.

Another way in which radioactivity can be used is to employ radioactive reagents in analytical procedures. The cost of such radioactive reagents has decreased in recent years to the point where they can be considered for use in analysis, especially on a micro scale. One example of this technique would be use of barium chloride containing radioactive barium to precipitate sulfate in a sultur determination. The insoluble barium sulfate can be separated and determined by detection for radioactivity with greater precision than is possible gravimetrically or volumetrically.

## APPLICATION TO PETROLEUM TESTING

From the foregoing discussion it can be seen that the entire field of petroleum analysis is substantially covered, test by test, by microprocedures that are closely equivalent to the standard large scale tests. These micro procedures can thus be used for obtaining the same information from small samples that is normally obtained by macro test procedures requiring much larger quantities of sample.

Of course, caution must be used when interpreting values obtained from micro tests based upon empirical test procedures since perfect correlation seldom is obtained. Due regard must also be paid to the lower degree of precision that is obtained when very small samples are tested.

### Product Analysis

Micro procedures are seldom used as control tests for petroleum products. Their greatest use is for identifying small amounts of oils present as films on equipment or for oil contained in small isolated pockets. Identifying oil in contaminated manufactured products and oil present in deposits and sludges removed from various sections of equipment is also of importance.

No general scheme of analysis is followed, since so many sample variations are possible. Three tests, viscosity, ash and qualitative spectrographic analysis, are often sufficient to solve the problem satisfactorily. If further testing is necessary, the information gained from these tests will probably suggest other procedures. The neutralization number is often of aid for detecting excessive oxidation of the oil. Quantitative elemental analysis of the additive elements can tell if an additive has been excessively depleted.

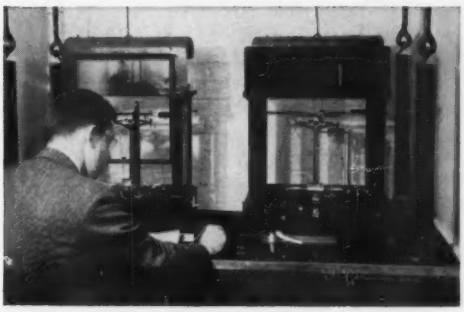


Figure 15 — Sensitive balances used for micro analysis are freed from building vibrations by being mounted upon a heavy steel table suspended from springs.

The micro analyst is greatly aided in his work by accurate reports of the conditions under which the lubricant had been used. He can then make an intelligent guess as to what contaminants to test for or what particular property of the oil will be selectively affected. A rough estimate of the value of a reliable field report would be to place its worth as equivalent to eight hours of the microanalyst's test time.

## Deposit Analysis

In addition to analyzing used oil samples, petroleum laboratories frequently have occasion to analyze samples of sludge and deposits taken from various parts of lubrication systems of engines and other equipment. When such analyses are interpreted correctly, it is possible to determine the cause of the deposit formation and indicate the lubrication problem. In many instances, the amount of material available for analysis is extremely small. It may consist of only a few milligrams of substance removed from a bearing, a piston or an oil cooler. Where such small samples are encountered, use of micromethods described previously are indispensable.

Deposits and sludges vary in physical appearance from a dry, metallic deposit to a soft, pasty sludge. There is no set pattern for analysis of all deposits. The analysis may be very limited or quite extensive depending on the individual case.

A microscopic examination of the sample under the low powers of a binocular microscope should be made in all instances. By picking over the sample with a spatula, specific materials such as metallic particles, rust, fibers and other foreign matter can be identified. A small magnet is used to determine qualitatively the amount of magnetic material in

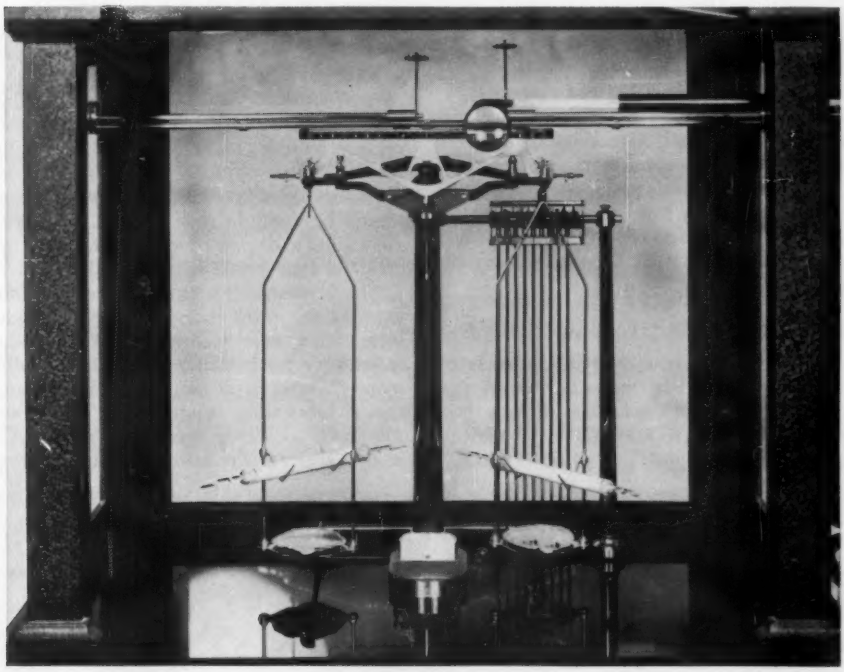


Figure 16 — Micro balance weighing absorption tube from carbon hydrogen determination.

the deposit. If finer structures are present in the sample, it may be desirable to make an examination under the high powers of a chemical microscope.

A micro ash content and spectrographic analysis is run on practically every deposit to be analyzed. In some cases it may be the only tests necessary on the deposit outside of microscopic examination.

A small glass centrifuge tube is used in the quantitative determination of the solubility of a deposit in pentane and benzene-methyl ethyl ketone. A sample of 100 milligrams or less is weighed into a centrifuge tube. Solvent is added and mixed with the sample. The tube is centrifuged in a small-scale centrifuge and the clear supernatant solvent is transferred to a micro beaker. More solvent is added, and the sample extracted several additional times until the supernatant solvent is colorless and free from dissolved matter. After drying, the residues in the centrifuge tube and micro beakers are weighed.

The pentane soluble material from the solubility separation of a deposit is the oily component. A few inspection tests on this fraction will indicate what type of oil was used in the system and whether contamination has occurred in cases where the oil used is known. Among the properties of the oil determined frequently are specific gravity, viscosity and pour point. Since the sample available is very small, the use of micro methods is imperative.

The benzene-methyl ethyl ketone soluble fraction or oxidized products is tested for metal soaps by ashing and determining the metal present spectrographically. The metal soap is decomposed by acid and the acidic fraction separated and examined.

The benzene-methyl ethyl ketone insoluble material or carbonaceous and mineral matter is given the same type of microscopic examination as the original deposit. The amounts of carbon and mineral matter are determined by a micro carbon and hydrogen analysis. X-ray diffraction analysis is frequently used to furnish identification of specific inorganic compounds present.

## RESEARCH AND DEVELOPMENT

The development of new chemical products by research is greatly aided by micro techniques that can be applied to the small quantities of substances initially produced. By screening new developments at this early stage, savings in both time and money can be obtained. The preparation of large amounts of experimental materials can be limited to those which micro analysis indicates to be favorable.

Under this heading one can also include the separation and identification of small quantities of additives from complex petroleum products. These mixtures can often be resolved by solvent extraction, chromatographic separations, distillation or sublimation. Micro analysis with strong assists from the instrumental methods is capable of identifying these fractions.

## SUMMARY

Micro analytical test procedures have a definite place in the modern petroleum analytical laboratory. Normally problems that have no other means of solution can be solved effectively by information that can only be obtained through micro analytical techniques. Reduction in the magnitude of operations frequently permits analyses to be carried out more rapidly and with more certain results than with conventional analytical procedures. When intricate apparatus is required for a determination, it is often easier and more economical to construct the unit on a micro scale. Reactions can be controlled more easily on a micro scale, and in cases where some safety hazard may exist, the use of micro techniques can materially reduce the hazard. Economy of operation is effected by micro techniques in cases where the required reagents are very expensive.

In those cases where micro techniques are used of necessity due to limited amounts of sample, the selection of procedures and order of testing must be planned carefully. Those test procedures which will allow substantial quantities of the sample to be recovered for further testing should be applied first. Insofar as it is possible, only those procedures expected to aid substantially in solving the problem should be used. In this regard, it is necessary in many cases to employ procedures indicated by results obtained from previous tests on the samples.

The future holds promise of more difficult problems for the micro analyst. Petroleum products are increasing in type and complexity. The equipment to which these products are applied is also being more complex with smaller assemblies and more critical lubricating requirements. These factors result in an increasing number of problems for the analyst, many of which require micro techniques.



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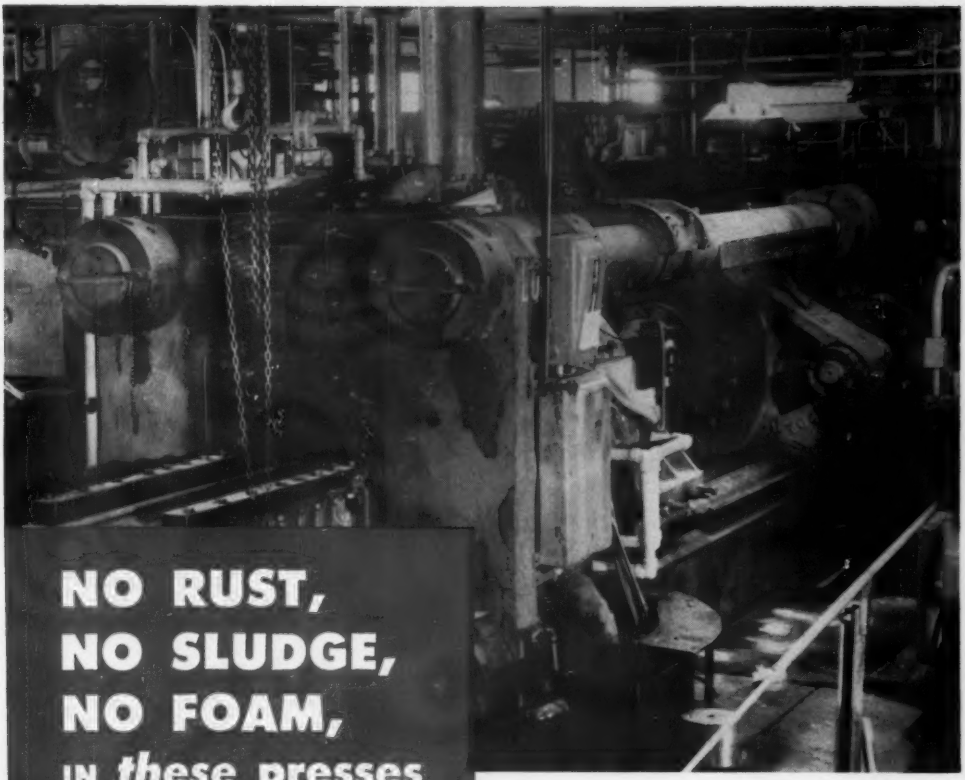

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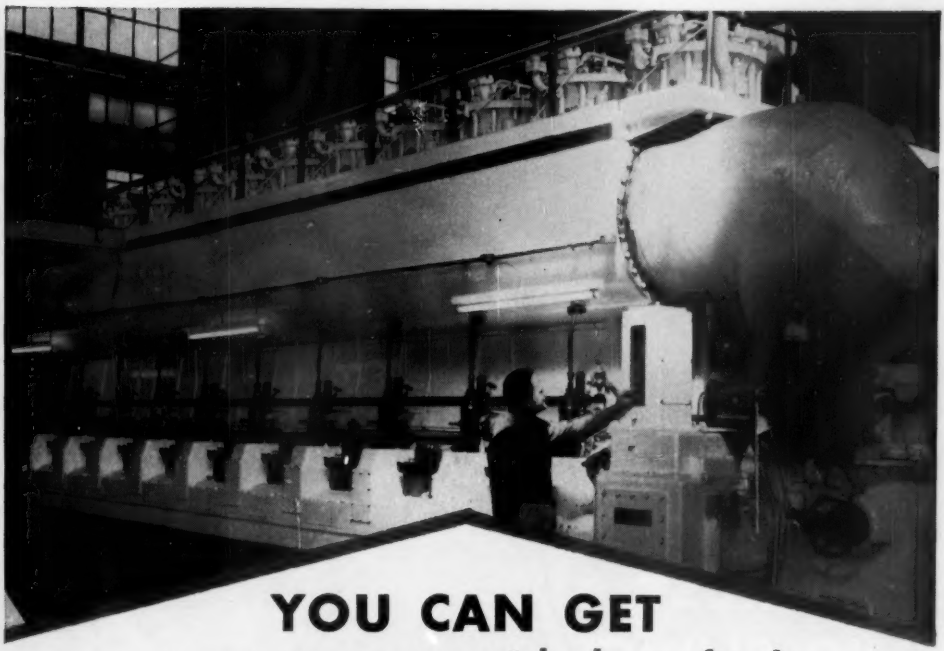

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